

Molecularly Oriented Polymeric Thin Films for Space Applications

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ABSTRACT

The increased commitment from NASA and private industry to the exploration of outer space and the use of orbital instrumentation to monitor the earth has focused attention on organic polymeric materials for a variety of applications in space. Some polymeric materials have exhibited short-term (3-5 yr) space environmental durability; however, future spacecraft are being designed with lifetimes projected to be 10-30 years. This gives rise to concern that material property change brought about during operation may result in unpredicted spacecraft performance. Because of their inherent toughness and flexibility, low density, thermal stability, radiation resistance and mechanical strength, aromatic polyimides have excellent potential use as advanced materials on large space structures. Also, there exists a need for high temperature (200-300°C) stable, flexible polymeric films that have high optical transparency in the 300-600nm range of the electromagnetic spectrum. Polymers suitable for these space applications were fabricated and characterized. Additionally, these polymers were molecularly oriented to further enhance their dimensional stability, stiffness, elongation and strength. Both unoriented and oriented polymeric thin films were also cryogenically treated to temperatures below -184°C to show their stability in cold environments and determine any changes in material properties.

KEY WORDS: Molecular Orientation, Colorless Polyimides, Space Applications, Cryogenic Treatment

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1. INTRODUCTION

The increased commitment from NASA and private industry to the exploration of outer space and the use of orbital instrumentation to monitor the Earth has focused attention on organic polymeric materials for a variety of applications in space. Certain high performance polymers offer attractive features such as low density, high strength, optical transparency and low dielectric constant. Polymeric matrix composites also offer weight savings, high strength, stiffness and dimensional stability. Some polymeric materials have exhibited short-term (3-5 yr) space environmental durability; however, future spacecraft are being designed with lifetimes projected to be 10-30 years. This gives rise to concern that material property change brought about during operation may result in unpredicted spacecraft performance[1].

The importance of lightweight, space environmentally durable polymers for applications on large deployable and inflatable structures was brought to the forefront in the spring of 1996 when NASA successfully deployed the Inflatable Antenna Experiment (14 meter lenticular structure)[2-3] from the Space Shuttle. Both industry and NASA recognized that this technology could support future missions that require very large aperture antennas, sunshades, concentrators, solar arrays, etc. Successful deployment of the Inflatable Antenna Experiment also demonstrated the feasibility that structures too large for mechanical deployment and too costly for in-orbit construction could be achieved by utilizing a film based inflatable design.

Utility of Langley Research Center (LaRC) developed thin polymeric films has been proposed for the Next Generation Space Telescope (NGST) sunshield. The NGST (Figure 1) is designed to use imaging and spectroscopy in the infrared to study the first stars and galaxies that formed after the universe cooled sufficiently to permit discrete structures to form. One of the conceptual components on the NGST is a large, multi-layer sunshield deployed by inflation, to shade all parts of the spacecraft over a reasonable range of pitch and roll, and reach telescope temperatures less than -213°C (-243°C goal). Polymeric thin films would be useful in this application due to their light weight, high strength, stiffness, dimensional stability, resistance to space environmental effects, and their ability to be metallized or coated[4-7]. Although the long term performance of polymeric thin films at cryogenic temperatures has not been determined, cryogenic treatment of metallurgical materials has been demonstrated to enhance the life of surface coatings and lengthen the life span of machine tools[8-13]. The same principles of enhancement may work with non-metallic materials, such as carbon-carbon composites, fibers and polymers as well.

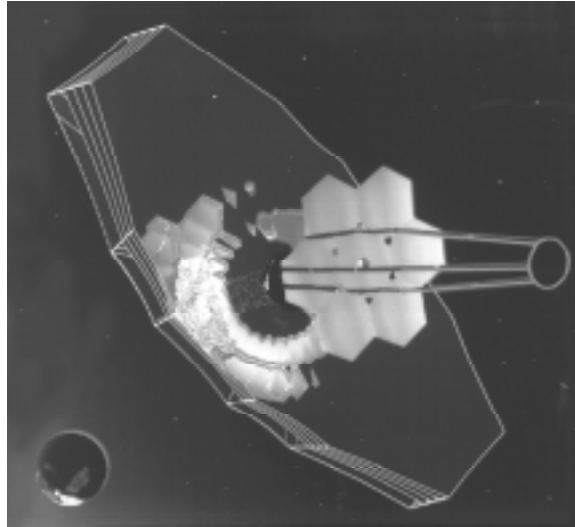


Figure 1 The Conceptual Design of the Next Generation Space Telescope (NGST)

In addition to polymers for large deployable and inflatable structure applications, the advent of space environmentally durable polymers is also expected to lead to other high payoff technologies. These include adhesives for spacecraft components, matrix resins for structural composites, multi-layer thermal insulators (MLI), solar array substrates, antenna reflectors/collectors, membranes for inflatable structures, and thermal control coating systems.

Molecular orientation and cryogenic treatment have been utilized in this study to potentially enhance material properties of space durable polyimides. Polymer film properties influenced by stretching include but are not limited to [14-25]: (1) mechanical properties such as tensile strength, modulus and elongation, (2) structural properties such as orientation, crystallinity and birefringence, and (3) other properties such as electrical conductivity, coefficient of thermal expansion, creep and surface roughness. Some general trends observed when films are oriented: (1) tensile strength and modulus increase with increased stretch ratio, (2) elongation and creep strain decrease with increased stretch ratio, (3) crystallinity increases with increased stretch ratio and stretching rate, and (4) birefringence increases with increased stretching rate and stretch ratio but decreases with increased stretching temperature. Stretching parameters such as stretching rate, stretch ratio, and temperature influence the mechanical, physical, and structural properties of polymers to various degrees. Temperature is a key element in optimizing properties by stretching.

Cryogenic treatment has improved material properties in metals. It is used to harden machine tools and enhance the life of surface coatings. It is proposed that the degree of freedom of molecules and atoms in molecular and atomic structures is deeply frozen under cryogenic temperature and consequently the bonding energy between the molecules or atoms is maximized and generates a marginally stable phase. In such a transition, materials experience slow but firm transformation of molecular and atomic structures. To date, not much research

has been done on the cryogenic treatment of nonmetallic materials such as carbon-carbon composites, fibers and polymers.

The overall research objectives will investigate the effects of molecular orientation (stretching) and cryogenic treatment of space durable polyimides for potential enhancement of material properties. Planned work will include the effects of: thermally versus chemically imidized films, different solvents and cure cycles, different cryogenic treatments, uniaxial versus biaxial stretching, and different polyimide backbone structures. The work presented herein will focus on the preliminary results of films fabricated from chemically imidized polyimide powders. Films were uniaxially stretched at various stretch ratios and cryogenically treated by slowly cooling to -184°C . Changes in material properties due to molecular orientation and cryogenic treatment are presented.

2. EXPERIMENTAL

2.1 Starting Materials The following chemicals were obtained from the indicated sources and used without further purification: 1,3-bis(3-aminophenoxy) benzene (APB, National Starch and Mitsui Toatsu), 2,2-bis[4-(4-aminophenoxy)phenyl] hexafluoropropane (4-BDAF, Chriskev), 2,2-bis(3,4-dicarboxyphenyl) hexafluoropropane dianhydride (6FDA, Hoechst Celanese), N,N-dimethylacetamide (DMAc, Aldrich, HPLC grade), acetic anhydride (Fisher Scientific, ACS grade), and pyridine (Aldrich, HPLC grade).

2.1.1 Polyimide Films The poly(amide acid) (PAA) solutions were prepared at a concentration of 15% solids (w/w) by the addition of a stoichiometric amount of the dianhydride in powder form to a mechanically stirred solution of the diamine in DMAc under a nitrogen atmosphere at room temperature (RT). PAA solutions were stirred 16-24 hr. The PAA solutions were chemically imidized using a mixture of pyridine/acetic anhydride. Three times the molar amount of both pyridine and acetic anhydride was added to the PAA solution and the solution was stirred an additional 16-24 hr at RT. The polyimide was then precipitated by pouring the chemically imidized PAA solution into a blender of distilled water. The powder was washed several times with distilled water, collected, and dried in a flowing air oven for 15-17 hours at 115°C and subsequently dried for 3 hr at 200°C under vacuum. Films were fabricated by dissolving the chemically imidized polyimide powder in DMAc (15% solids) and removing the solvent using the following heating cycles: (1) 50, 100 and 150°C for 1 hr at each temperature, (2) 100 and 200°C for 1 hr at each temperature, (3) 100, 200 and 300°C for 1 hr at each temperature.

2.1.2 Molecular Orientation Films were uniaxially stretched using a T. M. Long Film Stretcher. The T. M. Long Film Stretcher is a commercially available apparatus for uniaxial or biaxial stretching of polymer films. The film stretcher operates by the movement of two draw bars at right angles to each other upon hydraulically driven rods. A fixed draw bar is

located opposite each moving draw bar. Together, they make up the two axes at right angles along which the specimen is stretched. The stretch ratio is up to four times the original size. Film may be stretched in one or both directions independently, or in both directions simultaneously. The constant stretch rate can be varied from 0.508 to 50.8 cm per minute. The constant force can be varied from 0 to 0.17 MPa. Nominal specimen size is 10.1 x 10.1 cm between grips. Specimens are cut oversized to 11.2 x 11.2 cm to ensure the maximum gripping surface area for the film specimen. There are 52 clips (13 on each draw bar) to hold the specimen in place. Each clip has a miniature air cylinder. The clips are connected to a high pressure nitrogen source. The film is heated from both sides by streams of high velocity heated air from air heaters. The sample is placed on a vacuum plate and the loader is run in and dropped down to load the film. The loader is retracted and the cover lowered for stretching.

2.1.3 Cryogenic Treatment Cryogenic treatment was performed at 300° Below Inc. (Decatur, IL). The cryogenic treatment cycle was 48 hr. Samples were cooled slowly from RT to -184°C over a period of approximately 12 hr. Samples were held at -184°C for 24 hr and then slowly warmed to RT over a nominal 12 hr period.

2.1.4 Other Characterization Inherent viscosities (η_{inh}) of polyimide powders were obtained on 0.5% (w/v) solutions in DMAc at 35°C. Differential scanning calorimetry (DSC) was conducted on a Seiko DSC Model 210 thermal analyzer at a heating rate of 20°C/min with the T_g taken at the inflection point of the ΔT versus temperature curve. Dynamic thermogravimetric analyses (TGA) were performed on a Seiko TGA Model 5200 instrument at a heating rate of 2.5°C/min in air at a flow rate of 50 cm³/min. Coefficients of thermal expansion (CTE) were obtained on a Seiko TMA Model 100 at a heating rate of 2°C/min. The CTE values were determined between 70-125°C because of the linearity of the curve. Thin film tensile properties were measured as recommended in ASTM D882, using a SINTECH 2000-2 Computer Integrated Testing system, at ambient temperature and humidity. Crosshead speed was 0.508 cm/min; gage length was 5.08 cm. Pneumatically actuated one inch grips with padded steel faces were used for clamping the polyimide films. Films were cut with a 0.508 cm Thwing-Albert JDC Precision Cutter. Specimen thicknesses were determined to within 0.0000254 cm using a TMI Model No. 49-60 precision micrometer. Thickness measurements were made every inch over the specimen length and the average was used for the calculations. Refractive indices were determined using a Metricon® 2010 Prism Coupler. Refractive indices were measured in the plane of the sample in the stretch direction, in the plane of the sample at 90° to the stretch direction, and normal to the sample surface. The in-plane birefringence was calculated by the difference in refractive indices in the stretch direction and 90° to the stretch direction.

3. RESULTS AND DISCUSSION

Two colorless space durable polyimides, LaRC™-Colorless Polyimide 1 (LaRC™-CP1) and LaRC™-Colorless Polyimide 2 (LaRC™-CP2), were synthesized. Chemical structures of LaRC™-CP1 and LaRC™-CP2 are shown in Figures 2 and 3. Inherent viscosities of polyimide powders ranged from 1.1 to 1.3 dL/g. Films were cast from DMAc at 0.076 to 0.089 cm thick and placed in a dust-free, humidity controlled chamber until tack-free. The solvent was removed by heating at various temperatures. Orientation is generally facilitated when there is solvent left in the film since it acts like a plasticizer. Films were uniaxially stretched and cryogenically treated after orientation to determine any changes in material properties. Properties of control films (unstretched) for each batch of polymer films were used to determine changes in material properties because all the solvent was not removed in all stretching studies.

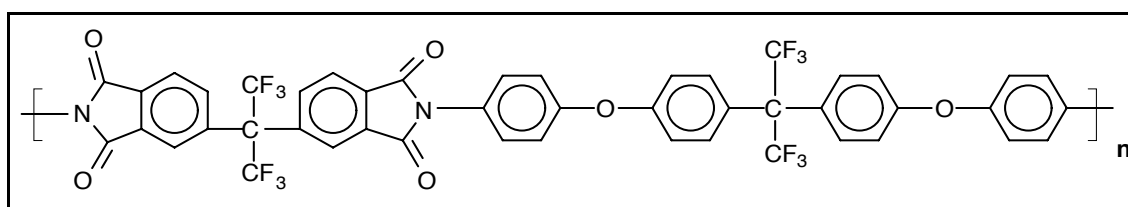


Figure 2. Chemical Structure of LaRC™-CP1

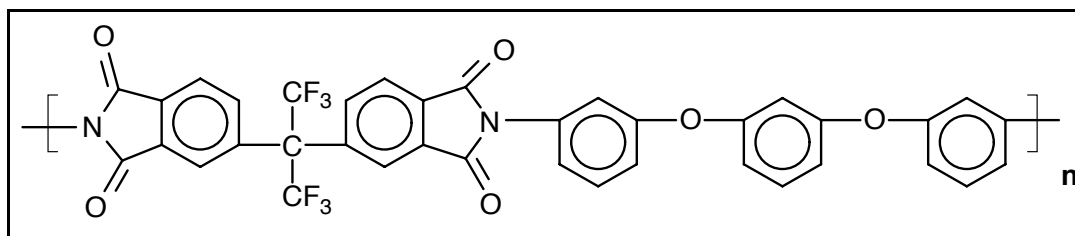


Figure 3. Chemical Structure of LaRC™-CP2

Films were stretched uniaxially using a T. M. Long Stretcher at ratios of 1.75X, 2X, 2.25X, 2.5X, 2.75X and 3X the original length of the samples. Successful stretching of samples depended on film quality, film thickness, soak time, grip pressure, stretching rate, and stretching temperature. Defects (particulate matter, air bubbles, etc.) in film samples resulted in tearing. Films of nonuniform thickness and thin films tore prematurely. Soak times ranged from 30 to 60 sec depending on film thickness. Grip pressures ranged from 1.03 to 2.07 MPa. If the grip pressure was too high, the films were more susceptible to tearing near the grips during orientation. If pressure was too low, films could not be restrained during orientation. Optimum grip pressure was approximately 1.6 MPa for samples ranging in thickness from 0.00762 cm to 0.0127 cm. Samples were stretched at rates ranging from 0.254 cm/sec to 0.508 cm/sec. Optimum stretching rate correlated with 0.46 to 0.62 MPa hydraulic pressure reading on the draw bar. The general trend observed for stretching temperature was a minimum of 50°C above the T_g of the polymer. Stretching temperatures for the LaRC™-CP1 polyimide films ranged from 310-340°C and stretching temperatures for the LaRC™-CP2 films ranged from 250-280°C. LaRC™-CP1 films were not stretched at all

stretch ratios because of higher temperature requirements and more difficulty encountered in the orientation process. The T. M. Long Stretcher operates more effectively at temperatures below 300°C because of the potential degradation of the o-rings in the actuators and the heating elements supplying the stretching temperature. Additional stretching temperatures will be attempted using LaRC™-CP1 films in future research efforts.

Table 1 shows physical properties of LaRC™-CP2 polyimide films heated at various temperature cycles (highest temperature indicated in column) and stretched at different ratios. The T_gs of the unstretched films and films heated to less than 300°C for solvent removal were low; solvent remained in the films; T_gs of second runs are indicated in parenthesis. Stretched films heated to 300°C exhibited T_gs ranging from 204 to 208°C which is consistent with fully imidized films without solvent. Higher stretch ratios were obtained in films that had only been heated to 150°C but data presented later in the paper will show that films heated to 300°C can be stretched to high ratios as well. The temperature at which 10% weight loss occurred increased with increased stretching ratios as expected since solvent removal was facilitated with stretching at high temperatures (250 to 280°C). CTE decreased with increased stretching ratios. The largest decrease in CTE and lowest value was obtained with films heated to 300°C and stretched at 2.25X.

Table 1. Characterization of Stretched LaRC™-CP2 Polyimide Film

Solvent removal cycle	Stretch Ratio	T _g , °C	10% wt loss temp., °C	CTE, ppm/°C
150°C	none	131 (207)	464	54
150°C	2.25X	207	481	44
150°C	2.5X	143 (205)	472	40
150°C	2.75X	138 (204)	482	39
200°C	none	166 (205)	493	48
200°C	2.125X	208	499	39
300°C	none	203	496	45
300°C	2X	204	501	42
300°C	2X	201	504	36
300°C	2.25X	201	511	31

Table 2 shows mechanical properties of oriented LaRC™-CP2 films. Tensile strengths and elongations to break increased with increased stretch ratio. Tensile strengths increased 36 to 47% depending on the solvent removal cycle. Elongations at break increased 93 to 95% depending on the solvent removal cycle. Moduli did not change significantly.

Table 2. Characterization of Stretched LaRC™-CP2 Polyimide Films

Solvent removal cycle	Stretch Ratio	Tensile str., MPa	Modulus, GPa	Elong., %
150°C	none	93.7	3.1	4
150°C	2.25X	164.7	2.7	84
150°C	2.5X	177.1	2.9	83
150°C	2.75X	175.7	2.9	84
200°C	none	102.0	3.1	5
200°C	2.125X	159.2	2.9	76
300°C	none	117.1	2.9	6
300°C	2X	145.4	2.7	56
300°C	2X	177.1	2.7	80
210.2	2.25X	210.2	2.7	57

More LaRC™-CP2 films were fabricated using the 100, 200 and 300°C solvent removal cycle and stretched at ratios of 2X, 2.25X, 2.5X and 3X. The same general trends were observed in these stretched samples. The unstretched and stretched samples were cryogenically treated by slowly cooling to -184°C, holding at -184°C for 24 hr and slowly warming back to RT. CTE and mechanical properties of unstretched films did not change after the cryogenic treatment. Tensile strengths of stretched films increased 10-20% with cryogenic treatment while moduli and elongations at break did not change significantly.

Additional LaRC™-CP2 films were fabricated and stretched to optimize the stretching conditions. The 100, 200 and 300°C solvent removal cycle was utilized and films were stretched at ratios of 2X, 2.5X, 2.75X and 3X. Physical and mechanical properties of the stretched LaRC™-CP2 samples are shown in Table 3. CTEs ranged from 36 to 45 ppm/°C. CTEs decreased with increased stretching ratios. Refractive indices were measured in the stretch direction (parallel to the optical symmetry axis), 90° to the optical symmetry axis in the sample plane, and normal to the sample surface. Refractive indices for all samples ranged from 1.60 to 1.62. In-plane birefringence was calculated by the difference in the refractive index in the stretch direction and the refractive index 90° to the stretch direction. Values are shown in Table 3. In-plane birefringence increased with stretching but did not follow a general trend as the stretch ratios increased from 2X to 3X. Hardaker *et al.* characterized three-dimensional properties of thick, free standing polymer films using prism coupling. Properties of drawn pyromellitic-oxydianiline films were also reported[26-27]. Tensile strengths and elongations increased with increased stretch ratios. Tensile strength increased 39% and elongation increased 95% with a 3X stretch ratio. Modulus did not change significantly with stretching. The stretched samples were cryogenically treated by slowly cooling to -184°C, holding at -184°C for 24 hr and slowly warming back to RT. Tensile strengths and moduli were not significantly affected by the cryogenic treatment. Elongations at break decreased slightly in some stretched samples.

Table 3. Characterization of LaRC™-CP2 Stretched Films

Stretch Ratio	CTE, ppm/°C	In-plane Birefringence	Tensile str., MPa	Modulus, GPa	Elong., %
none	45	-0.0002	103.4	2.4	5
2X	44	0.0048	108.2	2.5	23
2X	45	-0.0718	112.3	2.8	5
2.5X	40	-0.1617	134.4	2.6	87
2.5X	37	0.0193	169.5	2.6	90
2.5X	38	0.0032	151.6	2.7	96
2.5X	39	0.0115	166.1	2.9	59
2.75X	36	0.0079	162.6	2.6	84
2.75X	41	0.0099	144.7	2.6	79
3X	35	0.0169	170.2	2.6	99

LaRC™-CP1 films were heated to 300°C to remove solvent. LaRC™-CP1 films were stretched at 1.5X, 1.75X and 2X. Higher stretch temperatures were required to stretch LaRC™-CP1 because of its high T_g. Table 4 shows physical and mechanical characterization of stretched LaRC™-CP1 films. CTEs decreased with stretching, but the greatest decrease in CTE was observed with a 1.5X stretch. Tensile strengths increased up to 36% with the highest increase in tensile strength resulting from a stretch ratio of 2X. Moduli decreased slightly with samples stretched less than 2X. Moduli did not change significantly with a 2X stretch. Elongations increased with increased stretch ratio. LaRC™-CP1 stretched films were cryogenically treated and subsequently characterized. The tensile strength of 1.5X stretched film increased 40% with cryogenic treatment while the tensile strength of the 2X film decreased 20% with cryogenic treatment. The tensile strength of the unstretched film and the 1.75X stretched film did not change significantly with cryogenic treatment. Modulus of the stretched films did not change significantly with cryogenic treatment. Elongations did not change significantly with cryogenic treatment.

Table 4. Characterization of Stretched LaRC™-CP1 Polyimide Films

Stretch Ratio	CTE, ppm/°C	Tensile str., MPa	Modulus, GPa	Elong., %
none	50	93.0	2.0	16
1.5X	42	88.9	1.7	20
1.75X	44	107.5	1.8	49
1.75X	46	92.3	1.8	42
2X	46	145.4	2.1	65

4. SUMMARY

LaRC™-CP1 and LaRC™-CP2 were stretched at ratios of 1.75X, 2X, 2.25X, 2.5X, 2.75X and 3X their original length to potentially enhance their physical and mechanical properties. Successful stretching of samples depended on film quality, film thickness, soak time, grip pressure, stretching rate, and stretching temperature. Stretched specimens were cryogenically treated by cooling slowly to -184°C to determine any changes in material properties. Glass transition temperatures and the temperature at which 10% weight loss occurred were unaffected by stretching unless there was solvent remaining in the films. In-plane birefringence increased with increased stretch ratios. CTEs decreased with increased stretch ratio, as much as 31%. Tensile strengths increased up to 44% with increased stretch ratio. Elongations increased as much as 95% with stretching. Moduli did not change significantly with stretching. Cryogenic treatment increased the tensile strength of the some stretched samples approximately 10-20%. Other properties such as modulus and CTE did not change significantly after being slow cooled to cryogenic temperatures. Future studies will compare stretching of thermally imidized and chemically imidized films, evaluate different solvent and cure cycles, investigate the effects of different cryogenic treatments, fabricate uniaxial films with additional stretch ratios and initiate biaxial stretching of films, and orient other polyimides useful in aerospace, space, and microelectronics applications.

5. ACKNOWLEDGMENT

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The use of trade names of manufacturers does not constitute an official endorsement of such products or manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.

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7. BIOGRAPHIES

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Anne K. St. Clair is currently Chief Scientist of the Materials Division at NASA Langley Research Center, Hampton, VA. Her research interests include the synthesis, characterization and development of high performance polymer films, coatings and fibers for use on advanced aircraft and spacecraft.